

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

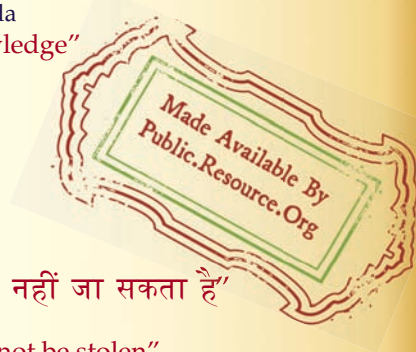
IS 14384 (1996): Colloidal graphite dispersion in oil [PCD
3: Petroleum, Lubricants and their Related Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



IS 14384 : 1996

भारतीय मानक

(Reaffirmed 2002)

तेल में कोलाइडल ग्रेफाइट परिक्षेपण — विशिष्टि

Indian Standard

COLLOIDAL GRAPHITE DISPERSION
IN OIL — SPECIFICATION

ICS 75.100

© BIS 1996

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1996

Price Group 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Lubricants and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Graphite is a solid lubricant particularly useful in high temperature lubrication for running of machinery and mechanisms, where an idle machine residual lubricant film containing fine graphite would prevent seizure on starting. Colloidal graphite dispersions in oil - light, medium and heavy are widely used in defence, shipping, machine tools, glass and heavy engineering industries.

Considerable assistance has been derived from CS 1419C, IND/SL/4116(a) Specification and Defence Standard 91-30/Issue 1, 12 July 1977 as well as the specifications of leading manufacturers of such material while preparing this standard.

It was the intention of the Committee to specify quality requirements of colloidal graphite powder used in the standard. However the same has not been included as part of the standard since the test methods to check quality requirements are still to be standardized. Consequently the requirements of synthetic colloidal graphite powder are given below for information:

<i>Sl No.</i>	<i>Characteristics</i>	<i>Requirements</i>
(1)	(2)	(3)
i)	Purity — Carbon, percent by mass, <i>Min</i>	99.8
	Ash, percent by mass, <i>Max</i>	0.2
ii)	Crystalline Structure	
	Crystalline size, nm, <i>Min</i>	60
	Interlayer distance, nm, <i>Min</i>	0.335 9
iii)	Density	
	Xylene (g/cm ³)	2.258
	Scott (g/cm ³)	0.09
iv)	Specific Surface BET (m ² /g)	22
v)	Particle size, μm , <i>Min</i>	
	Laser Diffraction	
	Less than 16 μm , percent	100.0
	Less than 8 μm , percent	99.3
	Less than 4 μm , percent	68.0
	Less than 2 μm , percent	20.0
	Less than 1 μm , percent	8.5

However these requirements may be agreed to between the purchaser and the supplier.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

COLLOIDAL GRAPHITE DISPERSION IN OIL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for colloidal graphite dispersion in oil intended for use as an industrial lubricant for special applications such as glass mould release agents, plunger lubricants, penetrating oils, chain lubricants, oil decasting and lubrication of certain artillery and guarded weapon equipment.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire-cloth test sieves (<i>third revision</i>)
1447 : 1966	Methods of sampling of petroleum and its products
1448	Methods of test for petroleum and its products:
[P:2] : 1967	Acidity (<i>first revision</i>)
[P:4] : 1984	Ash, sulphated ash and water soluble ash (<i>second revision</i>)
[P:10] : 1970	Cloud point and pour point (<i>first revision</i>)
[P:25] : 1976	Determination of kinematic and dynamic viscosity (<i>first revision</i>)
[P:34] : 1979	Determination of sulphur in petroleum products (Lamp method) (<i>second revision</i>)
[P:51] : 1963	Copper strip corrosion test for lubricating greases

IS No

Title

[P:69] : 1969	Flash and fire point by Cleveland (Open) cup
1745 : 1978	Petroleum hydrocarbon solvents

3 TYPES

There shall be three types of colloidal graphite dispersion in oil depending upon the type of oil used:

i) Light, ii) Medium, and iii) Heavy.

4 REQUIREMENTS

4.1 General

The material shall be homogeneous, greyish black fluid, free from water, grit, foreign matter and other visible impurities.

4.2 Composition

The material shall consist of a stable suspension of fine graphite powder in refined mineral lubricating oil.

4.2.1 The material shall be prepared from the following ingredients.

4.2.1.1 Synthetic colloidal graphite powder approximately 10 ± 0.5 percent by mass content.

4.2.1.2 Mineral lubricating oils (remainder)

The mineral oil used in the manufacture of graphite lubricant shall be refined mineral oil conforming to the requirements given in Table 1 when tested as per col 6 of Table 1.

4.2.1.3 Suitable wetting agents and dispersants may be incorporated for homogeneous stable dispersion of colloidal graphite in mineral oil.

4.3 The material shall also comply with the requirements given in Table 2, when tested according to the methods prescribed in col 6 and 7 of Table 2.

Table 1 Requirements of Mineral Oil

(Clause 4.2.1.2)

Sl No.	Characteristics	Types of Oils			Method of Test, Ref to P: of IS 1448
		Light (VG 100) (3)	Medium (VG 150) (4)	Heavy (VG 200) (5)	
(1)	(2)				(6)
i)	Viscosity, cSt at 40°C	90-110	135-165	200-240	P:25
ii)	Viscosity Index, <i>Min</i>	90	80	80	—
iii)	Flash point, COC, <i>Min</i>	200	200	200	P:69
iv)	Sulphur content, percent by mass, <i>Max</i>	1.20	1.50	1.70	P:34
v)	Pour point, °C, <i>Max</i>	-3.0	0.0	0.0	P:10

Table 2 Requirements for Colloidal Graphite in Oil

(Clause 4.3)

Sl No.	Characteristics	Requirements			Method of Test, Ref to	
		Light (3)	Medium (4)	Heavy (5)	(P:) of IS 1448 (6)	Annex of this Standard (7)
(1)	(2)					
i)	Kinematic viscosity at 40°C, cSt	90-110	135-165	200-240	P:25	—
ii)	Flash point (COC), °C, <i>Min</i>	200	200	200	P:69	—
iii)	Pour point, °C, <i>Max</i>	0.0	0.0	0.0	P:10	—
iv)	Acidity, inorganic (mg,KOH/g), <i>Max</i>	0.02	0.02	0.02	P:2	—
v)	Ash, percent by mass, <i>Max</i>	0.25	0.25	0.25	P:4	—
vi)	Copper strip corrosion for 3 h at 100°C, <i>Max</i>	← Not worse than No. 1 →			P:51	—
vii)	Visible residue	No visible residue			—	A
viii)	Size of graphite particle :				—	B
	a) Greater than 5 µm in diameter, percent, <i>Max</i>	2	2	2		
	b) Between 20 and 60 µm in diameter, <i>Max</i>	300	300	300		
	c) Greater than 60 µm in diameter, <i>Max</i>	NIL	NIL	NIL		
ix)	Stability of the suspension	← To pass the test →			—	C

5 KEEPING PROPERTIES (SHELF LIFE)

The material shall retain all the properties described under 4 of this specification for a period of at least one year from the date of manufacture when stored in its original sealed container under cover and at ambient temperature conditions not exceeding 50°C.

6 PACKING AND MARKING

6.1 The material shall be packed in suitable clean and dry leak-proof containers as agreed to between the purchaser and the supplier.

6.2 The containers shall be marked with the following:

- Name and type of the material;
- Net mass in the container;
- Indication of the source of manufacture;

- Recognized trade-mark, if any;
- Batch No. or Code No.; and
- 'SHAKE OR STIR WELL BEFORE USE'.

6.3 BIS Certification Marking

Each container may also be marked with the Standard Mark.

6.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447:1966.

ANNEX A

[Table 2, Sl No. (vii)]

DETERMINATION OF VISIBLE RESIDUE

(Adapted from DEF STAN 91-30/1)

A-1 OUTLINE OF THE METHOD

A-1.1 The sample is diluted with white spirit, and filtered through a test sieve. The sieve is then washed with further white spirit, dried in an oven and examined for evidence of visible residue.

A-2 APPARATUS

A-2.1 **Oven** — air drying capable of being maintained at $105 \pm 2^\circ\text{C}$.

A-2.2 **Beaker** — tall form, 150 ml.

A-2.3 **Tongs**

A-2.4 **Wire Cloth Test Sieve** — nominal aperture size 63 micron conforming to IS 460(Part 1):1985.

A-3 REAGENT

A-3.1 White spirit (*see* IS 1745:1978).

(Warning : White spirit is flammable. Keep away from sources of ignition).

A-4 PROCEDURE

A-4.1 Weigh 50 g of the sample into the beaker, dilute with 100 ml of white spirit and filter the mixture through the test sieve.

A-4.2 Wash the sieve with white spirit until the washings are clear.

A-4.3 Place the sieve in the oven maintained at $105 \pm 2^\circ\text{C}$ and dry for 15 minutes.

A-4.4 Using the tongs, remove the sieve from the oven and cool to room temperature.

A-4.5 Examine the sieve for any sign of residue.

A-5 REPORTING

A-5.1 Report whether or not any residue is visible on the sieve.

ANNEX B

[Table 2, Sl No. (viii)]

DETERMINATION OF PARTICLE SIZE OF GRAPHITE

(Adapted from DEF STAN 91/30 — Issue 1)

B-1 OUTLINE OF THE METHOD

B-1.1 The sample is stirred and then diluted with liquid paraffin. A drop of diluted suspension is examined under a microscope and the number of graphite particles, graded for size, determined.

B-2 APPARATUS

B-2.1 Mechanical Stirrer

B-2.2 Microscope — capable of resolving particles of 0.3 µm diameter, fitted with a mechanical stage.

B-2.3 Beakers — tall form, 150 ml.

B-2.4 Spatula

B-3 REAGENTS

B-3.1 Liquid Paraffin

B-4 PROCEDURE

B-4.1 Determination of Number of Particle Above 5 µm Diameter

B-4.1.1 Using the mechanical stirrer mix the sample thoroughly.

B-4.1.2 Weigh 0.2 ± 0.0001 g (to an accuracy of 0.1 mg) of the mixed sample into a beaker, add 20 ml of liquid paraffin and using the spatula mix thoroughly.

B-4.1.3 Transfer a drop of the diluted suspension to a cleaned and polished microscope slide and place a cover slip over it ensuring even distribution of the suspension and removal of air pockets.

B-4.1.4 Using the microscope, set to the appropriate magnification, count the number of particles (t) of diameter 0.3 µm or greater in a total area (a) of the slide ignoring any particles of diameter less than 0.3 µm.

NOTE — Use a total area (a) of the slide sufficiently large to contain at least 100 particles and consisting of at least 10 sub-areas chosen at random over the slides.

B-4.1.5 Using the same magnification, count also

the number of particles (g) greater than 5 µm in diameter in an area (b) of the slide, sufficiently large to contain at least 50 particles.

NOTE — If less than 50 particles have been counted when the area (b) is 100 times area (a), discontinue the count.

B-4.1.6 Calculate the percentage (P) of particles greater than 5 µm diameter as follows:

$$P = \frac{a \times g}{b \times t} - 100$$

when a , b , g and t have the usual significance.

B-4.2 Determination of Number of Particles Between 20 and 60 µm Diameter, and also Those Greater Than 60 µm Diameter

B-4.2.1 Using the mechanical stirrer, mix the sample thoroughly. Weigh 2 ± 0.0001 g (to an accuracy of 0.1 mg) of the mixed sample into a beaker, add 18 g of liquid paraffin (to an accuracy of 10 mg) and using the spatula mix thoroughly.

B-4.2.2 Transfer 4 mg of the diluted suspension (to an accuracy of 0.01 mg) to a cleaned and polished microscope slide and place the cover slip over, ensuring that the whole of the suspension is retained under it.

B-4.2.3 Using the microscope fitted with the 17 mm objective and x 15 eyepiece and by means of the mechanical stage scan successive strips of the microscope slide over the whole area of the suspension and count the total number of particles (a) between 20 and 60 µm diameter and (b) greater than 60 µm diameter.

B-5 REPORTING

B-5.1 Report the particle distribution of the graphite in the diluted suspension as follows:

- a) No. of particles greater than 5 µm diameter, percent;
- b) No. of particles between 20 and 60 µm diameter; and
- c) No. of particles greater than 60 µm diameter.

ANNEX C

[Table 2, Sl No. (ix)]

DETERMINATION OF STABILITY OF SUSPENSION

(Adapted from DEF STAN 91-30/T)

C-1 OUTLINE OF THE METHOD

C-1.1 Samples of the oil are centrifuged over a range of relative centrifugal force (rcf), drained under prescribed conditions and the residue remaining in the centrifuge tubes are weighed. A control, which is not centrifuged, is also drained and weighed. The retained weights of residue are plotted against rcf values to give a stability curve for the sample.

C-2 APPARATUS

C-2.1 Centrifuge — capable of maintaining a relative centrifugal force (rcf) of 80 000 at the tip of the tubes.

C-2.2 Centrifuge Tubes — plastic 20 ml.

C-2.3 Rack — for draining centrifuge tubes when inclined at 60° to the horizontal.

C-2.4 Mechanical Shaker**C-3 PROCEDURE**

C-3.1 Clamp the sample in its original container in the mechanical shaker and shake for 10 minutes.

C-3.2 Weigh six of the centrifuge tubes and into each of them weigh 10 ± 0.1 g portion of the sample.

C-3.3 Use one of these portions as a control, placing the tube in an inverted position in the draining rack and allow it to drain for 60 seconds.

C-3.4 Remove the control from the draining rack and carefully clean and dry it externally with absorbent paper.

C-3.5 Reweigh the control and record the weight of the residue to an accuracy of 0.01 g.

C-3.6 Centrifuge the other five tubes, separately, for 10 minutes each so that one tube is subjected to one of the following rcf levels:

- a) 4 000
- b) 15 000
- c) 30 000
- d) 60 000
- e) 80 000

NOTES

- 1 Use a tared counter balance tube as required by the centrifuge.
- 2 During the centrifuging control the temperature within the centrifuge at $20 \pm 1^\circ\text{C}$.
- 3 The period of centrifuging is timed from when the test speed corresponding to the required rcf has been reached.

C-3.7 Remove each tube immediately from the centrifuge, and subject them to the draining and weighing procedures described in C-3.3 to C-3.5.

C-4 CALCULATION

C-4.1 Prepare a graph as follows.

C-4.1.1 Plot the weight of each residue in the five centrifuged tubes against the corresponding rcf levels.

C-4.1.2 Plot the weight of the residue in the control tube against zero rcf.

C-4.1.3 Draw a smooth curve through the six points.

C-5 REPORTING

C-5.1 Use the curve to compare the stability of suspension of the sample with those of previous samples.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 4(401)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 323 01 31, 323 83 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :

Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002

{ 323 76 17
323 38 41

Eastern : 1/14 C. I.T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054

{ 337 84 99, 337 85 61
337 86 26, 337 91 20

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022

{ 60 38 43
60 20 25

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

{ 235 02 16, 235 04 42
235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093

{ 832 92 95, 832 78 58
832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR.
COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD.
JAIPUR. KANPUR. LUCKNOW. PATNA. THIRUVANANTHAPURAM.